METHOD 7191

CHROMIUM (ATOMIC ABSORPTION, FURNACE TECHNIQUE)

- 1.0 SCOPE AND APPLICATION
 - 1.1 See Section 1.0 of Method 7000.
- 2.0 SUMMARY OF METHOD
 - 2.1 See Section 2.0 of Method 7000.
- 3.0 INTERFERENCES
 - 3.1 See Section 3.0 of Method 7000 if interferences are suspected.
- $3.2\,$ Low concentrations of calcium and/or phosphate may cause interferences; at concentrations above 200 mg/L, calcium's effect is constant and eliminates the effect of phosphate. Calcium nitrate is therefore added to ensure a known constant effect.
- 3.3 Nitrogen should not be used as the purge gas because of a possible CN band interference.
- 3.4 Background correction may be required because nonspecific absorption and scattering can be significant at the analytical wavelength. Background correction with certain instruments may be difficult at this wavelength due to low-intensity output from hydrogen or deuterium lamps. Consult the specific instrument manufacturer's literature for details.
- 4.0 APPARATUS AND MATERIALS
 - 4.1 For basic apparatus, see Section 4.0 of Method 7000.
 - 4.2 Instrument parameters (general):
 - 4.2.1 Drying time and temp: 30 sec at 125°C.
 - 4.2.2 Ashing time and temp: 30 sec at 1000°C.
 - 4.2.3 Atomizing time and temp: 10 sec at 2700°C.
 - 4.2.4 Purge gas: Argon (nitrogen should not be used).
 - 4.2.5 Wavelength: 357.9 nm.
 - 4.2.6 Background correction: Not required.
 - 4.2.7 Other operating parameters should be set as specified by the particular instrument manufacturer.
 - NOTE: The above concentration values and instrument conditions are for a Perkin-Elmer HGA-2100, based on the use of a 20-uL injection,

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Revision 0 Date September 1986 continuous-flow purge gas, and nonpyrolytic graphite. Smaller sizes of furnace devices or those employing faster rates of atomization can be operated using lower atomization temperatures for shorter time periods than the above-recommended settings.

5.0 REAGENTS

5.1 See Section 5.0 of Method 7000.

5.2 <u>Preparation of standards</u>:

- 5.2.1 Stock solution: Dissolve 1.923 g of chromium trioxide (CrO $_3$, analytical reagent grade) in Type II water, acidify with redistilled HNO $_3$, and dilute to 1 liter. Alternatively, procure a certified standard from a supplier and verify by comparison with a second standard.
- 5.2.2 Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis. These standards should be prepared to contain 0.5% (v/v) HNO_3 ; 1 mL of 30% H_2O_2 and 1 mL of calcium nitrate solution, Section 5.2.3, may be added to lessen interferences (see Section 3.0).
- 5.2.3 Calcium nitrate solution: Dissolve 11.8 g of calcium nitrate, $Ca(NO_3)_2\cdot 4H_2O$ (analytical reagent grade), in Type II water and dilute to 1 liter.

6.0 SAMPLE COLLECTION. PRESERVATION. AND HANDLING

6.1 See Chapter Three, Section 3.1.3, Sample Handling and Preservation.

7.0 PROCEDURE

- 7.1 <u>Sample preparation</u>: The procedures for preparation of the sample are given in Chapter Three, Section 3.2.
- 7.2 See Method 7000, Paragraph 7.3, Furnace Procedure. The calculation is given in Method 7000, Paragraph 7.4.

8.0 QUALITY CONTROL

8.1 See Section 8.0 of Method 7000.

9.0 METHOD PERFORMANCE

9.1 Precision and accuracy data are available in Method 218.2 of Methods for Chemical Analysis of Water and Wastes.

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9.2 The performance characteristics for an aqueous sample free of interferences are:

Optimum concentration range: 5-100 ug/L. Detection limit: 1 ug/L.

9.3 The data shown in Table 1 were obtained from records of state and contractor laboratories. The data are intended to show the precision of the combined sample preparation and analysis method.

10.0 REFERENCES

- 1. Methods for Chemical Analysis of Water and Wastes, EPA-600/4-82-055, December 1982, Method 218.2.
- 2. Gaskill, A., Compilation and Evaluation of RCRA Method Performance Data, Work Assignment No. 2, EPA Contract No. 68-01-7075, September 1986.

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TABLE 1. METHOD PERFORMANCE DATA

Sample Matrix	Preparation Method	Laboratory Replicates
Paint primer	3050	2.7, 2.8 mg/g
Contaminated soil	3050	12.0, 12.3 ug/g
Oily lagoon soil	3050	69.6, 70.3 ug/g
NBS SRM 1646 Estuarine sediment	3050	42, 47 ug/gª
EPA QC Sludge	3050	156 ug/g⁵
NBS SRM 1085, Wear Metals in lubricating oil	3050	311, 356 ug/g ^c

 $^{^{\}rm a}{\rm Bias}$ of -45 and -38% from expected, respectively.

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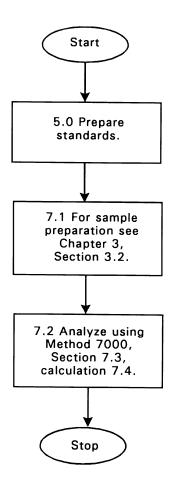
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^bBias of -24% from expected.

^cBias of +4 and +19% from expected, respectively.

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